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PATENT SPECIFICATION

NO DRAWINGS

1.122.756



Date of Application and filing Complete Specification: 25 July, 1967. No. 34045/67.

Application made in Germany (No. B88200 IVb/12q) on 27 July, 1966. Complete Specification Published: 7 Aug., 1968.

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In SC6 323 22Y 24 64, 84)

SPECIFICATION No. 1,122,756

Page 2, line 37, after "A" insert "comma"
Page 2, line 67, for "there" read "these"
Page 3, TABLE 1, second line of seventh compound delete "phenyl"
Page 5, line 46, delete whole line insert "tion of 1-[2-(3-diethylaminopropoxy-1)-"
Page 6, line 35, for "naphthyl)-6-carbinol"
read "naphthyl-6)-carbinol"
THE PATENT OFFICE

vatives of the present invention are compounds

23rd September 1968

R₁ X X O - A - Y

of the general formula:-

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(I)

wherein R₁ and R₂, which may be the same or different, are hydrogen or halogen atoms or nitro groups or alkyl, alkoxy or trifluoromethyl radicals, X is a straight-chain or branched alkylene or alkenylene radical which can be substituted by an oxo or hydroxyl group and —A—Y is a straight-chain or branched alkyl radical substituted by a tertiary amino group; and the acid-addition salts and quaternary ammonium compounds thereof.

We have found that the new compounds according to the present invention possess outstanding fungicidal and/or bactericidal properties

As examples of tertiary amino groups Y, there may be mentioned alkylated amino groups or the radicals of nitrogen-containing heterocyclic compounds, such as pyrrolidino, piper-

[Price 4. 6d.]

 R_2

(II)

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Ю

in which R_1 , R_2 and X have the same meanings as above, is reacted with a compound of the general formula:—

$$U - A - Z \qquad (III) \qquad 45$$

in which A is a straight-chain or branched alkylene radical, U is a hydroxyl group or a hydroxyl group esterified with a reactive acid residue (as hereinafter defined) and Z is either the tertiary amino group Y is a group which can be converted into the tertiary amino group Y, whereafter, if necessary, the group Z is converted, in known manner, into the tertiary amino group Y; or

b) an amino-alkoxy-naphthalene derivative 55 of the general formula:—

SEE ERRATA SLIP ATTACHED

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(I)

Index at acceptance:—C2 C(1E4K4, 1E7D2, 1E7E1, 1E7F2, 1E7N5, 1F2C4, 1F2C5, 1F2C6, 1F2D2, LF22X, LF32Y, LF36Y, LF43X, LF50Y, LF220, LF226, LF323, LF364, LF509, LF652, LF662, LM22X, LM29X, LM29Y, LM36Y, LM220, LM226, LM306, LM323, LM351, LM355, LM364, LM650, LM660); A5 E(1C4A2, 1C4A3, 1C4A4, 1C4B2, 1C4B3, 1C4B4)

Int. Cl.:—C 07 c 87/02, C 07 d 29/12, C 07 d 87/28

COMPLETE SPECIFICATION

Amino-Substituted Naphthalene Derivatives

C. F. BOEHRINGER & SOEHNE G.M.B.H., of Mannheim-Waldhof, Germany, a Body Corporate organised under the laws of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

The present invention is concerned with new amino-substituted naphthalene derivatives and with the preparation thereof.

The new basic-substituted naphthalene derivatives of the present invention are compounds of the general formula:-

wherein R₁ and R₂, which may be the same or different, are hydrogen or halogen atoms or nitro groups or alkyl, alkoxy or trifluoromethyl radicals, X is a straight-chain or branched alkylene or alkenylene radical which can be substituted by an oxo or hydroxyl group and —A—Y is a straight-chain or branched alkyl radical substituted by a tertiary amino group; and the acid-addition salts and quaternary ammonium compounds thereof.

We have found that the new compounds according to the present invention possess outstanding fungicidal and/or bactericidal proper-

As examples of tertiary amino groups Y, there may be mentioned alkylated amino groups or the radicals of nitrogen-containing heterocyclic compounds, such as pyrrolidino, piper-[Price 4. 6d.]

idino, morpholino, piperazino, and imidazolidino radicals.

The new compounds according to the present invention can be prepared in known manner. Thus, for example, either:

a) a naphthol derivative of the general formula: -

(II)

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in which R1, R2 and X have the same meanings as above, is reacted with a compound of the general formula:

$$U - A - Z$$
 (III) 45

in which A is a straight-chain or branched alkylene radical, U is a hydroxyl group or a hydroxyl group esterified with a reactive acid residue (as hereinafter defined) and Z is either the tertiary amino group Y is a group which can be converted into the tertiary amino group Y, whereafter, if necessary, the group Z is converted, in known manner, into the tertiary amino group Y; or

b) an amino-alkoxy-naphthalene derivative 55 of the general formula: -

(IV)

SEE ERRATA SLIP ATTACHED

in which —A—Y has the same meaning as above, is condensed with a compound of the general formula:—

in which R₁, R₂, X and U have the same meanings as above, whereafter, in the case in which X in the compound (I) obtained is an alkylene radical substituted by an oxo group, this is, if desired, subsequently reduced in known manner to the corresponding hydroxyl group-containing compound, this then, if desired, dehydrated to give the corresponding alkenylene compound which can, if desired, be reduced to give the corresponding alkylene compound and, if desired, the compounds (I) obtained can be converted into their acidaddition salts or quaternary ammonium compounds.

The reaction of compounds (II) and (III) takes place according to the usual methods of etherification, expediently by heating the reaction components in a suitable solvent in the presence of an alkali, an alkali metal alcoholate or an alkali metal amide.

When U is a hydroxyl group esterified with a reactive acid residue, it is either a halogen atom or an aromatic or aliphatic sulphonic acid radical, such as a p-toluene-sulphonyl, methyl-sulphonyl or p-bromobenzene-sulphonyl radical.

Instead of the aminoalkyl compounds U—A—Z, there can also be used the alkyl compounds U—A—U, such as alkyl dihalides, or the corresponding alkylene oxides, whereby there are obtained intermediates of the general

35 formula:—

(VI)

in which R₁, R₂, A U and X have the same meanings as above, which can subsequently be converted into compounds (I) by reaction with bases of the general formula H—Y, in which Y has the same meaning as above. When U in the compounds (VI) is a free hydroxyl group, this is expediently first converted into a reactive ester group (as hereinbefore defined) before the amination.

The condensation of compounds (IV) and (V) is carried out under the conditions of a Friedel-Crafts reaction, i.e. the reaction components are reacted in the presence of an acid or of a Lewis acid, such as aluminium chloride, and expediently also in the presence of an inert solvent.

When X in the starting materials (II) or (V) is an alkylene radical substituted by an oxo group, this group can subsequently be reduced, in known manner, in the compounds (I) obtained to give the corresponding hydroxy compounds, i.e. those in which X is a hydroxyl group-substituted alkylene radical, a complex metal hydride, such as sodium borohydride or lithium aluminium hydride, preferably being

These hydroxyl group-containing compounds can be converted into the corresponding unsaturated alkylene compounds by means of the usual dehydrating agents, such as alcoholic hydrochloric acid, and there, in turn, can be converted into the corresponding saturated alkylene derivatives, preferably by catalytic hydrogenation.

The basic compounds according to the present invention can with the help of inorganic or organic acids, be converted in known manner into the corresponding salts, such as the hydrohalides. For the preparation of the quaternary ammonium compounds, the basic compounds (I) are reacted with the conventional quaternising agents, such as alkyl or aralkyl halides.

The naphthol derivatives (II) used as starting materials can be obtained, for example, by the reaction of alkoxy-naphthalenes with a compound of general formula (V) under the conditions of a Friedel-Crafts reaction, whereby the alkoxy group is simultaneously or subsequently dealkylated to give a free hydroxyl group.

The aminoalkoxy-naphthalene derivatives (IV) can be prepared by the reaction of naphthols with compounds of general formula (III) and, if necessary, subsequent conversion of the residue Z into the basic group Y.

The following Examples are given for the purpose of illustrating the present invention:—

Example 1.
2-diethylaminoethoxy-6-(4-chlorophenacetyl)-naphthalene.

2.3 g. sodium (0.1 mol) are dissolved in 250 ml. isopropanol and boiled for 10 minutes with 29.6 g (0.1 mol) 6 - (4 - chlorophenacetyl) - naphthol - (2). After subsequent cooling, 14.8 g. diethylaminoethyl chloride (0.11 mol) are added dropwise and the reaction mixture boiled under reflux for 3 hours. The precipitate is filtered off with suction and the solvent removed in a vacuum. Thereafter, the residue is taken up in ether and ethereal hydrochloric acid added thereto which precipitates

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out the hydrochloride of 2 - diethylaminoethoxy - 6 - (4 - chloro - phenacetyl) - naphthalene which can be recrytsallised from isopropanol. The yield is 29.5 g. (68.5% of

theory) and the compound has a melting point of 186—187°C.

The compounds set out in the following Table I are obtained in an analogous manner.

TABLE I

compound	m.p. of hydrochloride	yield
2-diethylaminoethoxy-6-benzoyl-naphthalene	210 — 211°	74%
2-diethylaminoethoxy-6-(4-chlorobenzoyl)- naphthalene	about 128° amorphous	81%
2-diethylaminoethoxy-6-phenacetyl-naphthalene 2-diethylaminoethoxy-6-(4-methoxybenzoyl)- naphthalene	185 — 187°	71%
2-(3-dimethylaminopropers	170 — 172°	75%
2-(3-dimethylaminon	188 — 189°	80%
2-(diethylaminoethorn) 6 52 44	189 — 190°	79%
2-(3-diethylaminoproporty 1) 6 44	150 — 151°	69%
2-[2-(4-methyl-piporidinal 1)	178 — 180°	71%
2-[2-(4-methyl-piperidinyl-1)-ethoxy]-6-(4-chloro-phenacetyl)-naphthalene	210 — 211°	83%
2-[2-(morpholino-4)-ethoxy]-6-(4-chloro- phenacetyl)-naphthalene	198199°	73%
2-[2-(piperidinyl-1)-ethoxy]-6-(4-chloro- phenacetyl)-naphthalene	217 — 219°	79%
-(2-diethylaminoethoxy)-6-(2-chloro- henacetyl)-naphthalene	182 — 183°	
-(2-diethylaminoethoxy)-6-(3,4-dichloro- henacetyl)-naphthalene		71%
•	176 — 178°	63%

EXAMPLE 2.
2-diethylaminoethoxy-6-(4-methoxy-benzoyl)-naphthalene.

42 g. 2 _ dierhylaminoethoxy - naphthalene hydrochloride (0.15 mol; m.p. 140—141°C.) are stirred for 8 hours at room temperature with 25.5 g. anisoyl chloride (0.15 mol) and 36 g. aluminium chloride (0.3 mol) in 250 ml. nitrobenzene. The reaction mixture is subsequently left to stand overnight, decomposed by the addition of ice and the nitrobenzene driven off by passing through steam. The reaction mixture is then extracted with ether

and the acidic aqueous layer rendered alkaline by the addition of a solution of sodium hydroxide, the base thereby being liberated and subsequently extracted with ether. The ethereal extract is evaporated and the residue distilled at oil pump vacuum. There are obtained 26.2 g. (51.5% of theory) 2 - diethylaminoethoxy - 6 - (4 - methoxy - benzoyl)-naphthalene with a boiling point of 240—250°C./0.3 mm.Hg.

The hydrochloride prepared therefrom in the usual manner, melts at 170—172°C.

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EXAMPLE 3. Phenyl-(2-diethylaminoethoxy-naphthyl-6)-

carbinol.

6 g . 2 - diethylaminoethoxy - 6 - benzoylnaphthalene (0.0177 mol), prepared in the manner described in Example 1, are boiled for 2 hours with 0.68 g. lithium aluminium hydride (0.0177 mol) in 50 ml. ether. The reaction mixture is subsequently decomposed by the addition of a saturated aqueous solution of

sodium chloride, the organic layer separated off, dried over anhydrous sodium sulphate and evaporated. After recrystallisation of the residue from petroleum ether (b.p. 100—140°C.), there are obtained 5 g. (811% of theory) phenyl - (2 - diethylaminoethoxy - naphthylphenyl - (3 - diethylaminoethoxy - diethylaminoethoxy - naphthylphenyl - (3 - diethylaminoethoxy - diethyla 6) - carbinol with a melting point of 82-83°C.

The compounds set out in the following Table II are obtained in an analogous manner:

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TABLE II

compound	m.p. of hydrochloride	yield
-chlorobenzyl-[2-(3-dimethylaminopropoxy-1)- haphthyl-6]-carbinol	203 — 204° (m.p. of base 152 — 154°)	79%
4-chlorobenzyl-(2-diethylaminoethoxy-naphthyl-6)- carbinol	161 — 163°	83%
4-chlorobenzyl-[2-(3-diethylaminopropoxy-1)- naphthyl-6]-carbinol	204 — 205°	75%
4-chlorobenzyl-[2-(4-methylpiperidinyl-1)-ethoxy- naphthyl-6]-carbinol	190 — 192°	71%
2-chlorobenzyl-(2-diethylaminoethoxy-naphthyl-6)-carbinol	159 — 160°	85%
4-chlorobenzyl-[2-(morpholino-4)-ethoxy-naphthyl-6]-carbinol	155 — 156°	95%

EXAMPLE 4.

1-[2-(3-dimethylaminopropoxy-1)-naphthyl-

6]-2-(4-chloro-phenyl)-ethylene.
3 g. 4 - chlorobenzyl - [2 - (3 - dimethylaminopropoxy - 1) - naphthyl - 6] - carbinol hydrochloride (0.0072 mol), prepared in the manner described in Example 3, are boiled for 1 hour in 30 ml. alcoholic hydrochloric acid.

After cooling, there crystallise out 2.5 g. (86.5% of theory) 1 - [2 - (3 - dimethylamino-propoxy-1) - naphthyl - 6] - 2 - (4 - chlorophenyl) - ethylene in the form of the hydrochloride, which has a melting point of 275—

The compounds set out in the following Table III are obtained in an analogous manner.

compound	m.p. of hydrochloride	vield
1-[2-(2-diethylaminoethoxy-1)-naphthyl-6]-2- (4-chlorophenyl)-ethylene	221 — 223°	87%
1-[2-(2-diethylaminoethoxy-1)-naphthyl-6]-2-(2-chlorophenyl)-ethylene	184 — 185°	
1-[2-(morpholino-4)-ethoxy-1)-naphthyl-6]-2- (4-chlorophenyl)-ethylene	287 — 289°	83% 91%
1-[2-(3-diethylaminopropoxy-1)-naphthyl-6]-2- (4-chlorophenyl)-ethylene	213 — 214°	79%
1-[2-(2-diethylaminoethoxy-1)-naphthyl-6]-2- (3,4-dichlorophenyl)-ethylene	224 — 225°	72%
1-[2-(2-diethylaminoethoxy-1)-naphthyl-6]-2- phenyl-ethylene	238 — 239°	86%

EXAMPLE 5. 2-diethylaminoethoxy-6-(4-chlorophenacetyl)-naphthalene methiodide.

9.9 g. 2 - diethylaminoethoxy - 6 - (4-chloro-phenacetyl) - naphthalene (0.025 mol), prepared in the manner described in Example 1, are boiled for 2 hours with 4.2 g. methyl iodide (0.03 mol) in 50 ml. acetone. Upon cooling, crystals separate out and these are subsequently filtered off with suction and recrystallised from methyl ethyl ketone. There are obtained 12.0 g. 2 - diethylaminoethoxy-6 - (4 - chloro - phenacetyl) - naphthalene methiodide (86.5)% of theory) with a melting point of 152—153°C.

In an analogous manner, by reaction with ethyl bromide there is obtained 2 - diethylaminoethoxy - 6 - (4 - chloro - phenacetyl)-20 naphthalene ethobromide in a yield of 56% of theory, which has a melting point of 176-177°C., and by reaction with propyl iodide there is othained 2 - diethylaminoethoxy - 6-(4 - chloro - phenacetyl) - naphthalene propiodide in a yield of 49% of theory, which has a melting point of 160—161°C.

> EXAMPLE 6. 2-(2-diethylaminoethoxy-1)-6-[2-(2-chloro-

phenethyl-1)]-naphthalene.

10.4 g. 1 - [2 - (2 - diethylaminoethoxy1) - naphthyl - 6] - 2 - (2 - chlorophenyl)ethylene hydrochloride (0.025 mol), prepared in the manner described in Example 4, are dissolved in 100 ml. methanol and, after the addition of 0.5 g. 5% palladium-charcoal, hydrogenated without the use of pressure. The

reaction mixture is subsequently filtered with suction, the filtrate evaporated, the residue taken up in isopropanol and ether added thereto which precipitates out, in 72% yield, the partially amorphous hydrochloride of 2-(2-diethylaminoethoxy-1)-6-[2-(2-chlorophenethyl - 1)] - naphthalene with a melting point of about 123°C.

In an analogous manner, by the hydrogenation of 1 - [2 - 3 - (diethylaminopropyl - 1)-naphthyl - 6] - 2 - (4 - chlorophenyl)-ethylene hydrochloride, there is obtained, in 83% yield, the hydrochloride of 2 - (3-diethylaminopropoxy - 1) - 6 - [2 - (4-chloro - phenethyl - 1)] - naphthalene when, after recrystallisation from isopropanol, has a melting point of 154-155°C.

WHAT WE CLAIM IS:-1. Amino-substituted naphthalene derivatives of the general formula:-

wherein R₁ and R₂, which may be the same or different, are hydrogen or halogen atoms or nitro groups or alkyl, alkoxy or trifluoro-methyl radicals, X is a straight-chain or branched alkylene or alkenylene radical which may be substituted by an oxo or hydroxyl group and —A—Y is a straight-chain or

branched alkyl radical substituted by a tertiary amino group; and the acid-addition salts and quaternary ammonium compounds thereof. 2. 2 - Diethylaminoethoxy - 6 - (4 - chlorophenacetyl) - naphthalene. 3. 2 - Diethylaminoethoxy - 6 - benzoylnaphthalene. 4. 2 - Diethylaminoethoxy - 6 - (4 - chlorobenzoyl) - naphthalene. 5. 2 - Diethylaminoethoxy - 6 - phenacetylnaphthalene. 6. 2 - Diethylaminoethoxy - 6 - (4 - methoxy - benzoyl) - naphthalene. 7. 2 - (3 - Dimethylaminopropoxy - 1)-- (4 - chloro - phenacetyl) - naphthalene. 8. 2 - (3 - Dimethylaminopropoxy - 1)-- (4 - chloro - benzoyl) - naphthalene. 9. 2 - (Diethylaminoaminoethoxy) - 6-[3 - (4 - chlorophenyl) - propionyl] - naphthalene. 10. 2 -(3 - Diethylaminopropoxy - 1) - 6-(4 - chloro - phenacetyl) -naphthalene. 11. 2 - [2 - (4 - Methyl - piperidinyl - 1)-ethoxy] - 6 - (4 - chloro - phenacetyl) - naphthalene. 12. 2 - [2 - (Morpholino - 4) - ethoxy]-6 - (4 - chloro - phenacetyl) - naphthalene. 13. 2 - [2 - (Piperidinyl - 1) - ethoxy]-6 - (4 - chloro - phenacetyl) - naphthalene. 14. 2 - (2 - Diethylaminoethoxy) - 6 - (2chloro - phenacetyl) - naphthalene. 15. 2 - (2 - Diethylaminoethoxy) - 6 - (3,4dichloro - phenacetyl) - naphthalene. 16. Phenyl - (2 - diethylaminoethoxynaphthyl) - 6 - carbinol. 17. 4 - Chlorobenzyl - [2 - (3 - dimethylaminopropoxy - 1) - naphthyl - 6] - carbinol. 18. 4 - Chlorobenzyl - (2 - diethylaminoethoxy - naphthyl - 6) - carbinol. 19. 4 - Chlorobenzyl - [2 - (3 - diethylaminopropoxy - 1) - naphthyl - 6] - carbinol. 20. 4 - Chlorobenzyl - [2 - (4 - methylpiperidinyl- 1) - ethoxy - naphthyl - 6]carbinol. 21. 2 - Chlorobenzyl - (2 - diethylaminoethoxy - naphthyl - 6) - carbinol.

22. 4 - Chlorobenzyl - [2 - (morpholino-4) - ethoxy - naphthyl - 6] - carbinol.

23. 1 - [2 - (3 - Dimethylaminopropoxy-1) - naphthyl - 61 - 2 - (4 - chlorophenyl) 1) - naphthyl - 6] - 2 - (4 - chlorophenyl)ethylene. 24. 1 - [2 - (2 - Diethylaminoethoxy - 1) naphthyl _ 6] - 2 _ (4 - chlorophenyl)-25. 1 - [2 - (2 - Diethylaminoethoxy - 1)-naphthyl - 6] - 2 - (2 - chlorophenyl)-26. 1 - [2 - (Morpholino - 4) - ethoxy - 1)naphthyl - 6] - 2 - (4 - chlorophenyl)-

27. 1 - [2 - (3 - Diethylaminopropoxy-1) - naphthyl - 6] - 2 - (4 - chlorophenyl)- naphthyl - 6] - 2 - (3,4 - dichlorophenyl)ethylene. 29. 1 - [2 - (2 - Diethylaminoethoxy - 1)-naphthyl - 6] - 2 - phenyl - ethylene. 30. 2 - Diethylaminoethoxy - 6 - (4-chloro - phenacetyl) - naphthalene methiodide.

31. 2 - Diethylaminoethoxy - 6 - (4-70 chloro - phenacetyl) - naphthalene ethobromide. - Diethylaminoethoxy - 6 - (4-32. 2 chloro - phenacetyl) - naphthalene propiodide. 75 33. 2 - (2 - Diethylaminoethoxy - 1) - 6-[2 - (2 - chloro - phenethyl - 1)] - naphtha-34. 2 - (3 - Diethylaminopropoxy - 1) - 6-[2 - (4 - chloro - phenethyl - 1)] - naphtha-35. Process for the preparation of compounds of the general formula given in claim 1, wherein a naphthol derivative of the general 85 formula: -

28. 1 - [2 - (2 - Diethylaminoethoxy - 1)-

in which R1, R2 and X have the same meanings as in claim 1, is reacted with a compound of the general formula:-

$$U - A - Z \qquad 90$$

in which A is a straight-chain or branched alkylene radical, U is a hydroxyl group or a hydroxyl group esterified with a reactive acid residue (as hereinbefore defined) and Z is the tertiary amino group Y or a group which can be converted into Y, whereafter, if necessary, the substituent Z is converted into the group

36. Process according to claim 35, wherein the reaction is carried out in a solvent in the presence of an alkali, alkali metal alcoholate or alkali metal amide.

37. Process for the preparation of compounds of the general formula given in claim 1, wherein an amino-alkoxy-naphthalene deri- 105 vative of the general formula:-

in which -A-Y has the same meaning as in

ethylene.

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claim 1, is condensed with a compound of the general formula:—

in which R_1 , R_2 and X have the same meaning as in claim 1 and U is a hydroxyl group or a hydroxyl group esterified with a reactive acid residue (as hereinbefore defined).

38. Process according to claim 37, wherein the reaction is carried out in an inert solvent in the presence of a Friedel-Crafts catalyst.

in the presence of a Friedel-Crafts catalyst.

39. Process according to any of claims 35—38, wherein, when the product obtained is a compound in which X is an alkylene radical substituted by an oxo group, this oxo group is subsequently reduced to a hydroxyl group to give the corresponding hydroxyl group-containing compound.

40. Process according to claim 39, wherein the reduction is carried out with a complex metal hydride.

41. Process according to claim 39 or 40, wherein the hydroxyl group-containing compound is reacted with a dehydrating agent to give the corresponding alkenylene compound.

42. Process according to claim 41, wherein the dehydration is carried out with alcoholic hydrochloric acid.

43. Process according to claim 41 or 42, wherein the alkenylene compound is reduced to give the corresponding alkylene compound.

44. Process according to claim 43, wherein the reduction is carried out by catalytic hydrogenation.

45. Process according to any of claims 35—44, wherein the product obtained is subsequently reacted with a quaternising agent to give the corresponding quaternary ammonium compound.

46. Process according to any of claims 35—44, wherein the product obtained is subsequently reacted with an acid to give the corresponding addition salt.

ponding addition salt.

47. Process for the preparation of compounds of the general formula given in claim 1, substantially as hereinbefore described and exemplified.

48. Compounds of the general formula given in claim 1, whenever prepared by the process according to any of claims 35—47.

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